



User Guide

QuadraSil™ Metal Scavengers





User Guide - QuadraSil™ Metal Scavengers

Contents	Page
Introduction	3
Product Availability	3
QuadraSil™ Range	4
Features and Appearance	4
Guidelines for Use	5
<i>Metal-Contaminated Solutions</i>	5
Metal Scavenging Applications	6
iron chloride	6
copper acetate	6
triphenylphosphinecobalt(III)chloride	7
copper acetylacetonate	7
palladium acetate	8
copper bromide	8
ROPAC™	9
silver acetate	10
Catalyst Scavenging Example	
Grubbs catalyst	10
Metal Recovery	11
Storage	11
Appendix: QuadraSil™ Selection Guide	



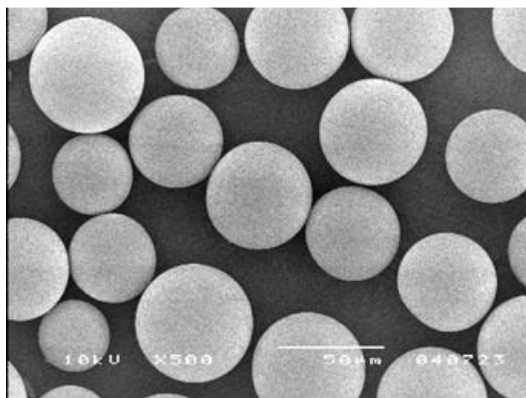
User Guide - QuadraSil™ Metal Scavengers

Introduction

The QuadraSil™ range of metal scavengers offers an attractive solution to metal contamination problems associated with pharmaceutical and fine chemical processing complementing the QuadraPure™ scavenger resin range. The QuadraSil™ products are functionalized silicas specifically designed for extraction of metal contaminants from organic and aqueous systems. As with the QuadraPure™ range, the very low levels of extractable impurities make products particularly suitable for GMP-compliant applications in both batch and continuous processing.

The QuadraSil™ series are ideal for use in R&D applications as they are extremely fast scavengers and easy to isolate by filtration. A QuadraSil™ metal selector guide is given in the Appendix.

Reaxa is continually developing the applications of its metal scavengers. The Company can also offer expertise in using all its metal-scavenger products in flow. Customer support in helping solve problems across the chemical industry sectors is a particular strength. For further information please contact Reaxa at info@reaxa.com.



microscope image of QuadraSil™

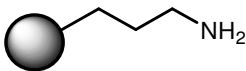
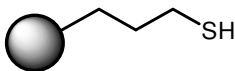
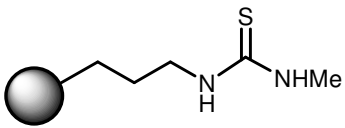
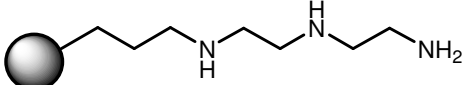
R&D quantities of QuadraPure™ and QuadraSil™ as well as test kits for screening are available from Sigma-Aldrich at:

www.sigma-aldrich.com

For technical support, bulk quotations and information please contact: info@reaxa.com

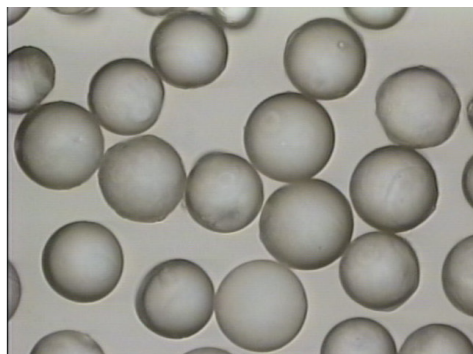
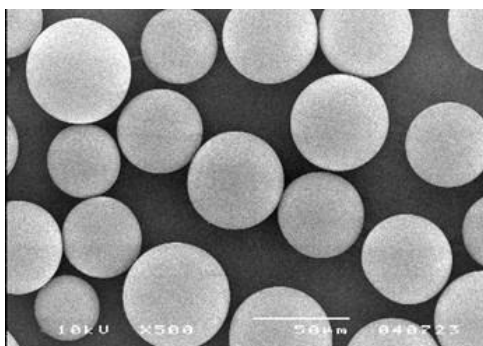


QuadraSil™ Range

QuadraSil™	functional group	mmol/g	Aldrich order #	
AP		amino propyl	2.0	679534
MP		mercaptopropyl	1.2	679526
MTU		methylthiourea	1.4	679518
TA		triamine	1.0	679496

Features and Appearance

- functionalised spherical mesoporous silicas
- compatible with organic, aqueous, protic and aprotic media
- extremely fast scavenging
- zero swelling characteristics ideal for use in fixed bed applications
- surface area 715 m²/g
- average particle size 54 µm
- mechanically and thermally robust



microscope images of QuadraSil™ highlighting the regularity of size and shape

The QuadraSil™ particles are highly robust and chemically resistant free-flowing silicas. The narrow particle size distribution, highly spherical shape, and zero swell makes these products suitable for use in fixed bed and cartridge format.



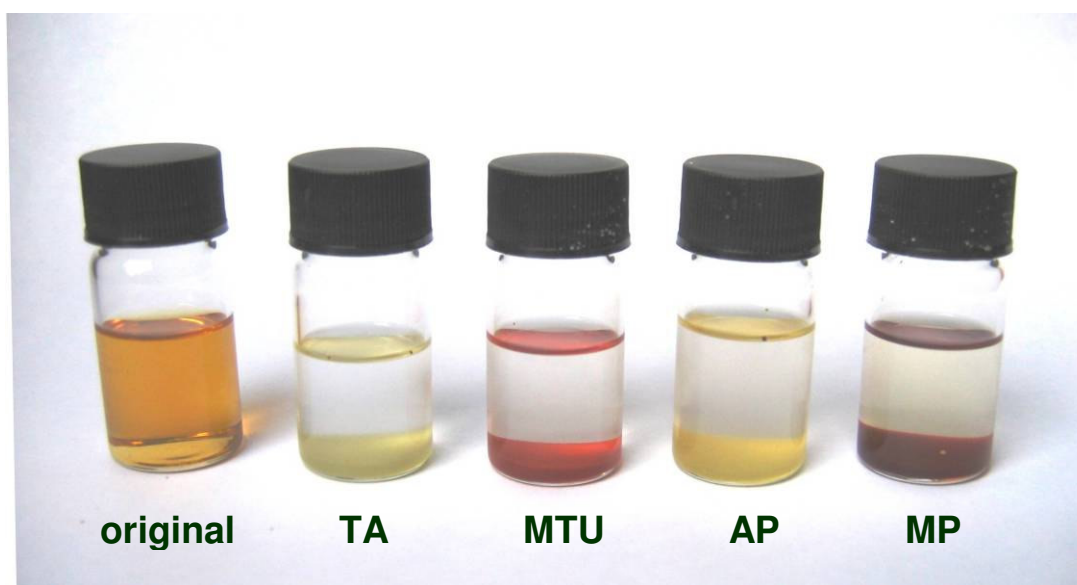
Guidelines for Use

Note: For further detailed user application notes, GMP regulatory support data, and advice on specific problems, please write to info@reaxa.com

Metal-Contaminated Solutions:

In metal contaminated organic solvents, direct addition of QuadraSil™ will effect scavenging. Typically, add 5 g per 100 ml metal-contaminated solution and leave to gently agitate at room temperature.

Metal removal is often complete in five minutes and can frequently be observed by a colour change in both the scavenger and the solution (see below). The resin is simply removed by filtration.

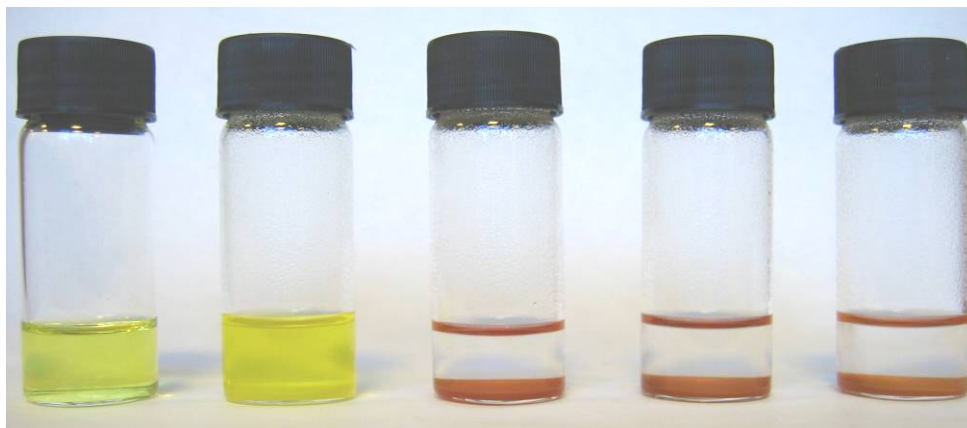
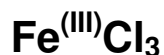


The rate of metal scavenging can be increased by the addition of more resin, raising the temperature or increasing the agitation rate.



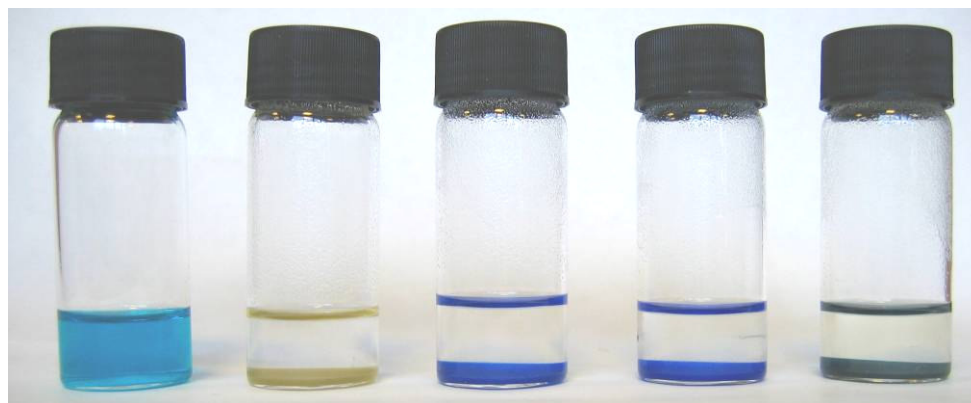
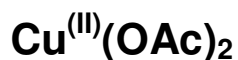
QuadraSil™ Metal Scavenging Applications

The following examples show simple trials for metal salt removal from common solvents. In each case the picture shows the original solution (far left) and then after treatment with the QuadraSil™ range at the loading recommended for initial testing (0.5 g per 10 ml). Times to clear the solution are recorded.



1000ppm solution of FeCl₃ in THF – 100ml solution: 291mg FeCl₃ in 100ml THF

	MP	AP	TA	MTU
Fe(III)Cl ₃	N/A	< 2 min	< 2 min	< 2 min

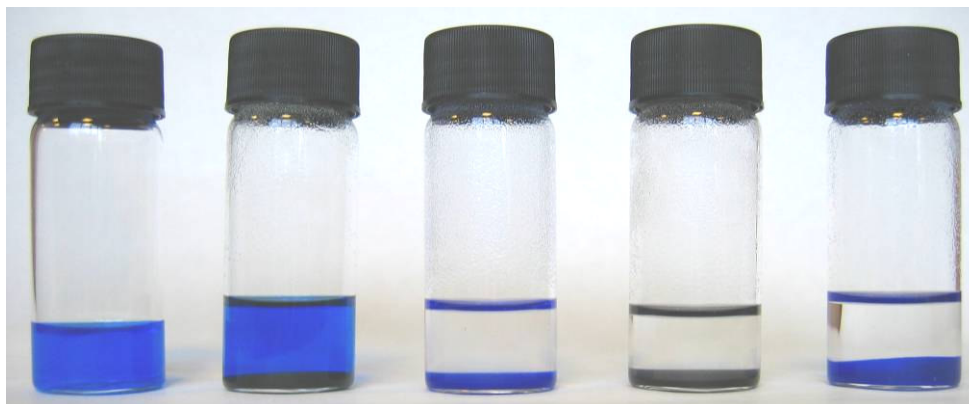


1000ppm solution of Cu(OAc)₂ in THF – 100ml solution: 258mg Cu(OAc)₂ in 100ml THF

	MP	AP	TA	MTU
Cu(II)(OAc) ₂	< 2 min	< 2 min	3 min	4 min



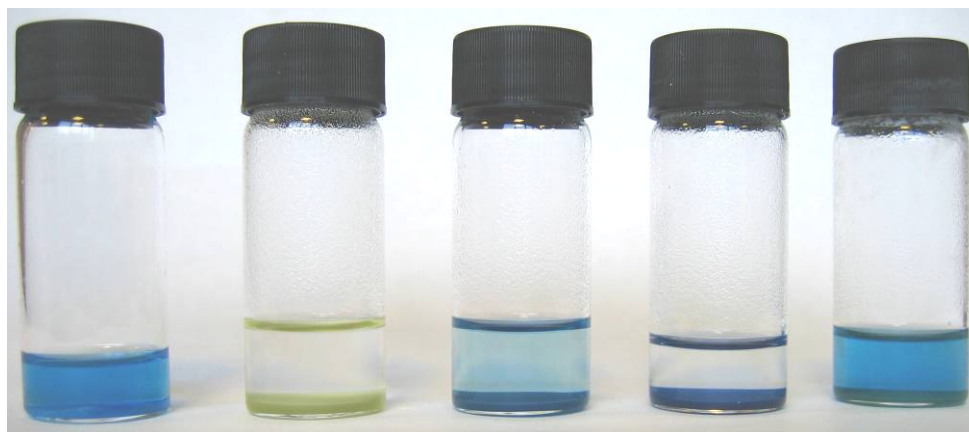
Co^(II)Cl₂(PPh₃)



1000ppm solution CoCl₂(PPh₃) in THF – 100ml solution: 1100mg CoCl₂(PPh₃) in 100ml THF

	MP	AP	TA	MTU
Co(II) dichlorobis-TPP	N/A	< 2 min	6 min	3 min

Cu^(II)(acac)₂

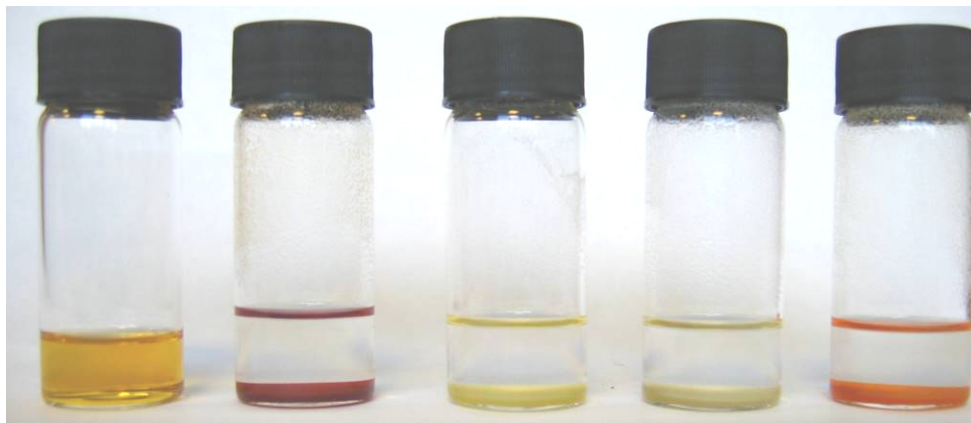


1000ppm solution Cu(acac)₂ in THF – 100ml solution: 412mg Cu(acac)₂ in 100ml THF

	MP	AP	TA	MTU
Cu ^(II) (acac) ₂	15 min	N/A	8 min	N/A



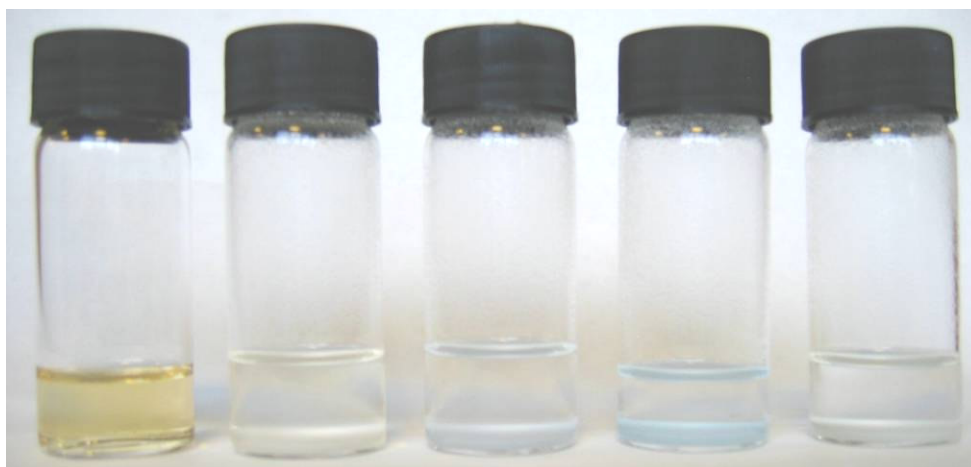
$\text{Pd}^{(II)}(\text{OAc})_2$



1000ppm solution $\text{Pd}(\text{OAc})_2$ in **DCM** – 100ml solution: 213mg $\text{Pd}(\text{OAc})_2$ in 100ml DCM

	MP	AP	TA	MTU
$\text{Pd}^{(II)}(\text{OAc})_2$	~1 min	~1 min	~1 min	~1 min

$\text{Cu}^{(I)}\text{Br}$

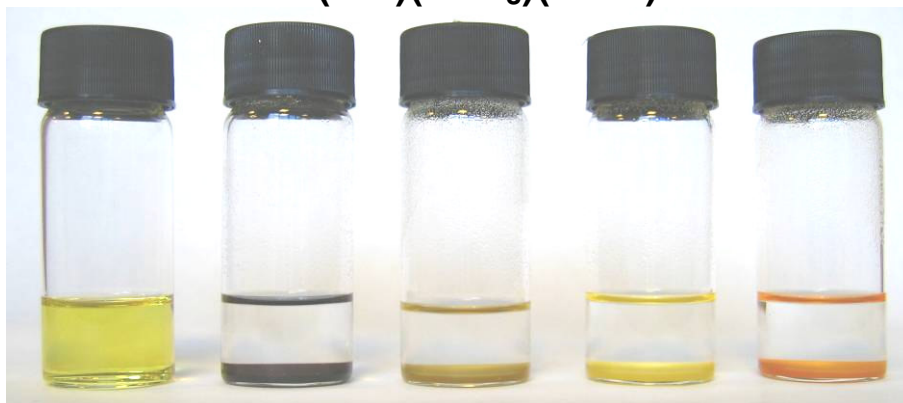


1000ppm solution CuBr in **THF** – 100ml solution: 213mg CuBr in 100ml THF

	MP	AP	TA	MTU
$\text{Cu}^{(I)}\text{Br}$	< 2 min	< 2 min	< 2 min	< 2 min



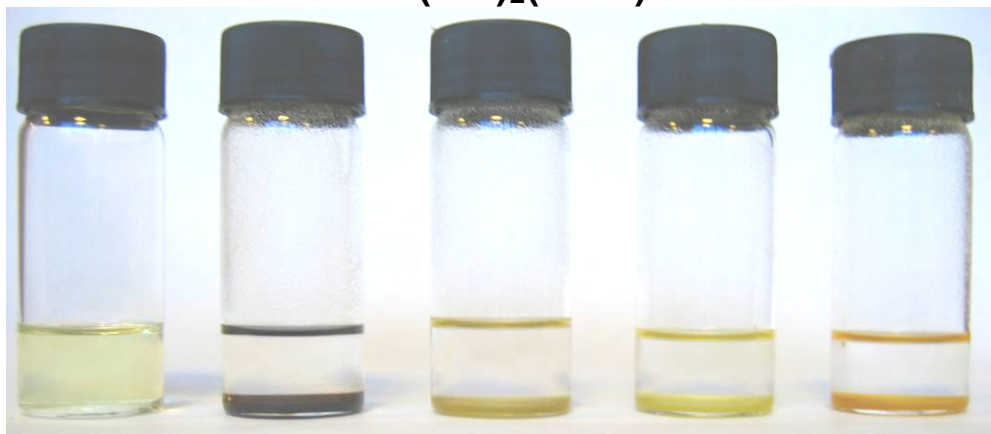
Rh(CO)(PPh₃)(acac)



1000ppm solution Rh(CO)(PPh₃)(acac) in **DCM** – 100ml solution: 481mg Rh(CO)(PPh₃)(acac) in 100ml DCM

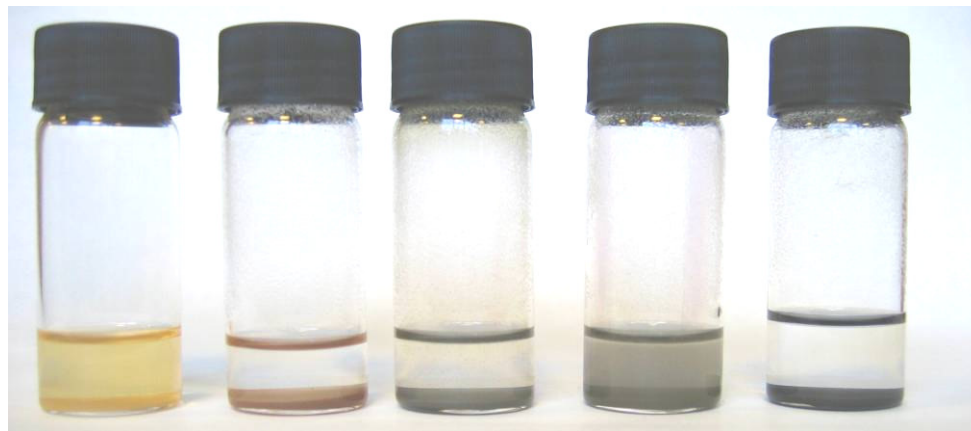
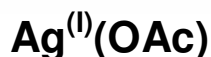
	MP	AP	TA	MTU
Rh(CO)(PPh ₃)(acac)	< 5 min	> 24 h	> 24 h	> 24 h

Rh(CO)₂(acac)



1000ppm solution Rh(CO)₂(acac) in **DCM** – 100ml solution: 239mg Rh(CO)₂(acac) in 100ml DCM

	MP	AP	TA	MTU
Rh(CO) ₂ (acac)	2 min	2 min	2 min	2 min



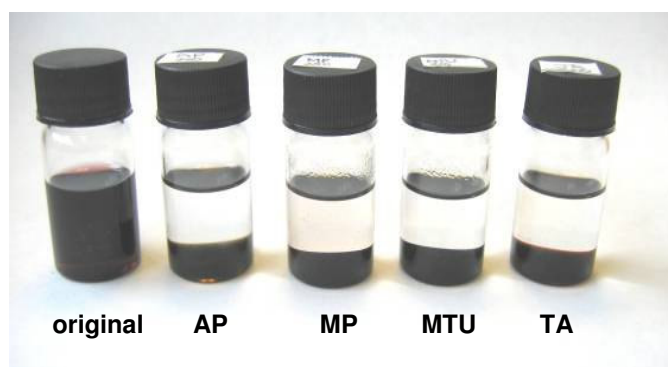
1000ppm solution Ag(OAc) in THF – 100ml solution: 155mg Ag(OAc) in 100ml THF

	MP	AP	TA	MTU
Ag(I)(OAc)	< 4 min	50 min	N/A	40 min

QuadraSil™ Catalyst Scavenging Example

Removal of ruthenium following metathesis using the Grubb's catalyst is a significant problem overcome by very few scavengers. The QuadraSil™ range, with AP in particular, show excellent scavenging ability for this catalyst. In the picture below the starting solution shown contains 595 ppm Ru. 5 ml aliquots were treated with 250 mg of each QuadraSil™. All reduced the levels to below 5 ppm, with QuadraSil™ AP clearing the solution in 1½ h.

QuadraSil™	initial Ru (ppm)	% Ru removal	Time (h)
AP	595	>99	1.5
MP	595	>99	<16
MTU	595	>99	<16
TA	595	>99	<16





Metal Recovery

Through Reaxa's partnership with Umicore we are able to offer complete metal management solutions to our customers.

Storage

The QuadraSil™ range of products are air-stable and can be stored at room temperature.



APPENDIX

QuadraSil™ Selection Guide

Please note that the following is a guide only and based on trials performed at Reaxa together with customer feedback. The efficiency of a metal scavenger will depend upon a number of factors including the oxidation state of the metal involved, pH and potential competing complexation by other ligands present and/ or the solvent. It is recommended that a range of QuadraPure™ and QuadraSil™ are assayed for each metal contamination problem.

	MP	AP	TA	MTU
Fe(III)	—	✓✓	✓✓	✓✓
Cu(II)	✓✓	✓✓	✓✓	✓✓
Cu(I)	✓✓	✓✓	✓✓	✓✓
Co(II)	—	✓✓	✓✓	✓✓
Ni(II)	✓	✓✓	—	—
Pd(II)	✓✓	✓✓	✓✓	✓✓
Pt	✓✓	—	✓✓	—
Ru(II)	✓	✓✓	✓	✓
Rh(II)	✓✓	✓✓	✓✓	✓✓
Ag(I)	✓✓	✓	—	✓

- ✓✓ Excellent Scavenging Ability (>99% removal, under 10 min)
- ✓ Significant Scavenging Ability (>98% removal, up to 24 h)
- Not Tested /No Significant Scavenging Ability